

Nitrate-Nitrite, Cadmium Reduction**SM 4500 NO₃⁻ E – 2000 (2011)**

ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 4020.

Facility Name: _____ LAB ID: _____

Assessor Name: _____ Analyst Name: _____ Inspection Date: _____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/Revision/Date: _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
If analyzing for Nitrite OR Nitrate, are samples preserved by storing at ≤6°C, and analyzed within 48 hours of collection?	40 CFR 136.3 Table 1I				
If analyzing for Nitrate-Nitrite, are samples preserved to pH <2 with sulfuric acid, preserved by storing at ≤6°C, and analyzed within 28 days of collection?	40 CFR 136.3 Table 1I				
Were sample results 0.01 – 1.0 mg NO ₃ ⁻ N/L?	NO ₃ ⁻ A - 1 Introduction				
Were turbid samples filtered through a 0.45-µm membrane filter?	NO ₃ ⁻ A - 1 Introduction				
Were filters tested for nitrate contamination?	NO ₃ ⁻ A - 1 Introduction				
Were samples for nitrate determination not acidified?	NO ₃ ⁻ A - 1 Introduction				
Were samples where copper, iron, and other metal concentrations above several mg/L was suspected treated with EDTA prior to analysis?	4500-NO ₃ ⁻ E 1 b				
Where samples were contaminated by oil and grease, were they solvent extracted prior to analysis?	4500-NO ₃ ⁻ E 1 b				
Were samples checked for residual chlorine, and, if RC was present, were samples treated with sodium thiosulfate?	4500-NO ₃ ⁻ E 1 b				
If a spectrophotometer was used, did it have a wavelength of 543 nm with a path length of 1 cm or longer?	4500-NO ₃ ⁻ E 2 b 1				
If a filter photometer was used, did it have a light path of 1 cm or longer and a transmittance near 540 nm?	4500-NO ₃ ⁻ E 2 b 2				
Did absorbances of reagent blanks not exceed 0.01?	4500-NO ₃ ⁻ E 3 a				
Notes/Comments:					

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Were 25 g aliquots of 20 to 100 mesh Cd granules washed with 6N HCl?	4500-NO ₃ ⁻ E 3 b				
In repeated cycles were Cd granules next swirled with 2% CuSO ₄ solution for either 5 minutes or until blue color faded until a brown colloidal precipitate developed?	4500-NO ₃ ⁻ E 3 b				
Were the nitrate solutions not used for longer than 6 months?	4500-NO ₃ ⁻ E 3 h				
Was the intermediate nitrite solution prepared daily?	4500-NO ₃ ⁻ E 3 k				
Did reduction columns first have glass wool plugs inserted into them, then get filled with water, and then have 18.5 cm of Cu-Cd granules added to them?	4500-NO ₃ ⁻ E 4 a				
Were reduction columns prepped by washing with 200 mL of dilute NH ₄ Cl-EDTA then about 100 mL of 25% 1.0 mg NO ₃ ⁻ -N/L+75% NH ₄ Cl-EDTA?	4500-NO ₃ ⁻ E 4 a				
Were sample pHs adjusted to be between 7 and 9 with dilute HCl or NaOH?	4500-NO ₃ ⁻ E 4 b 2				
Were 25 mL sample volumes mixed with 75 mL volumes of NH ₄ Cl-EDTA solution?	4500-NO ₃ ⁻ E 4 b 3				
Were the first 25 mL of sample NH ₄ Cl-EDTA solution mixtures that passed through the column discarded?	4500-NO ₃ ⁻ E 4 b 3				
If columns were not to be used for several hours or longer, were they stored with dilute NH ₄ Cl-EDTA solution and not allowed to dry?	4500-NO ₃ ⁻ E 4 b 3				
Was color reagent addition never more than 15 minutes after reduction?	4500-NO ₃ ⁻ E 4 b 4				
Were 2.0 mL of color reagent added to 50 mL volumes of reduced sample mixtures, and the absorbances measured against distilled water-reagent blanks at 543 nm?	4500-NO ₃ ⁻ E 4 b 4				
Were the absorbances of reduced sample mixtures measured between 10 minutes and 2 hours after color reagent addition?	4500-NO ₃ ⁻ E 4 b 4				
Was at least one NO ₂ ⁻ standard compared to a reduced NO ₃ ⁻ standard at the same concentration to verify column efficiency?	4500-NO ₃ ⁻ E 4 c				
Were columns reactivated when above column efficiency dropped below 75%?	4500-NO ₃ ⁻ E 4 c				
Was reduction of standards carried out exactly as described for samples?	4500-NO ₃ ⁻ E 4 c				
Notes/Comments:					